

B. Tech PROJECT REPORT ON

PREPARATION OF OXALIC ACID FROM MOLASSES

For partial fulfillment of the requirements for the degree of

Bachelor of Technology

in

Chemical Engineering

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This is to certify that the report on “**PREPARATION OF OXALIC ACID FROM MOLASSES**” submitted by **AMIT SETHY (109CH0030)** in partial fulfillment of the requirements for the award of degree of Bachelor of Technology in Chemical Engineering at National Institute of Technology, Rourkela is an authentic work carried out by him under my supervision and guidance.

To best of knowledge, the matter embodied in this thesis has not been submitted to any other university or institute for the award of any degree.

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ABSTRACT

Oxalic acid is the simplest of dicarboxylic acid. Its name is derived from the Greek Oxyes meaning sharp, acidic referring to the acidity common in the foliage of certain plants from which it was first isolated. Oxalic acid is commercially available as the dihydrate containing 28.5% water. It is a monoclinic prism, particles size varying from fine powder to coarse granules which are also colorless, melting point 187°C of anhydrous form and 101.5°C of dihydrate form. Oxalic acid is one of the most widespread organic acid in plants. In this project a commercial method of preparation of Oxalic acid at 30 ton per day is presented using molasses as the starting material. This project also highlights the material & energy balance as well as description of a few equipments used in this process. Other important features includes cost estimation , instrumentation& process control of equipment used in the process & a possible plant layout based on scientific principles & commercial requirement.

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NOMENCLATURE

FBR- Fluidized bed reactor

CSTR- continuous stirred tank reactor

ΔH = enthalpy

Kg- kilograms

Kj- kilojoules

CHAPTER – 1

INTRODUCTION

1. INTRODUCTION

1.1 OXALIC ACID

Oxalic acid is an organic compound with the formula $\text{H}_2\text{C}_2\text{O}_4$. It is a colorless crystalline solid that dissolves in water to give colorless solutions. It is classified as a dicarboxylic acid. In terms of acid strength, it is much stronger than acetic acid^[1] Oxalic acid is a reducing agent and its conjugate base, known as oxalate ($\text{C}_2\text{O}_4^{2-}$), is a chelating agent for metal^[1]

1.2 PHYSICAL & CHEMICAL PROPERTIES^[9]

Appearance... Transparent, colourless, odourless crystals.

Solubility... 1gm/7ml (water).

Specific Gravity... 1.65

Boiling Point... 149 - 160°C (sublimes).

Melting Point... 101.5°C.

Vapour Density... 4.4 (air=1).

Vapour Pressure... < 0.001 @ 20°C (mm Hg).

Molecular Weight-126.07.

Chemical Formula... $\text{HOOC}\text{COOH} \cdot 2\text{H}_2\text{O}$

Oxalic acid, is purchased usually in the form of oxalic acid dihydrate, which is a crystalline form with two water molecules attached to each molecule of oxalic acid. Oxalic acid is the simplest of dicarboxylic acid. Its name is derived from the Greek Oxeyes meaning sharp, acidic suffering to the acidity common in the foliage of certain plants from which it was first isolated. Oxalic acid is commercially available as

the dihydrate containing 28.5% water. Oxalic acid is odorless and white in colour. It is a monoclinic prism, with particles size varying from fine powder to coarse granules which are also colorless, melting point 187°C of anhydrous form. Oxalic acid is a normal metabolite of carbohydrates in normal quantities, e.g., Fructose, Glycine, and Vitamin C.

1.3 MOLASSES & ITS PROPERTIES^[2]

Molasses is a viscous by-product of the refining of sugarcane, grapes, or sugar beets into sugar. The word comes from the Portuguese “melaço”, ultimately derived from Mel, the Latin word for "honey". The quality of molasses depends on the maturity of the source plant, the amount of sugar extracted, and the method employed. Molasses are of various types based on plant material from which it is produced. So different types of molasses produced based upon various raw materials are cane molasses from sugarcane, beet molasses from sugar beet, grape molasses from grapes. Its major constituents are-

- 1) Glucose – 35.9%
- 2) Fructose - 5.6%
- 3) Sucrose – 2.6%
- 4) Water - 23.5%

Uses of Molasses

- Molasses is commonly used with yeast and water in the fermentation process of making rum. It is also used in creating some other alcoholic drinks such as stout and dark ales.
- Some tobacco companies add it to their product for smoking through a certain type of pipe popular in the Middle Eastern countries.

- Some anglers use molasses as a groundbait (also called chum or berley). This is designed to attract fish to the area that an angler is fishing.
- It can also be mixed with water and used to remove rust.
- It is used in some horticultural settings to promote microbe activity in the soil.
- Molasses contains trace amounts of important vitamins and minerals. A certain type of molasses called blackstrap molasses has been sold as a health supplement for many years. People have made many claims about its health benefits, including the ability to prevent gray hair.
- Molasses is also added to some cattle feed to add essential vitamins and minerals

Glucose a major component of molasses also known as D-glucose, dextrose, or grape sugar is a simple monosaccharide found in plants. It is one of the three dietary monosaccharides, along with Fructose and Galactose, that are absorbed directly into the bloodstream during digestion. An important carbohydrate in biology, cells use it as the primary source of energy and a metabolic intermediate. Glucose is one of the main products of photosynthesis and fuels for cellular respiration. Glucose exists in several different molecular structures, but all of these structures can be divided into two families of mirror-images (stereoisomers). Only one set of these isomers exists in nature, those derived from the "right-handed form" of glucose, denoted D-glucose. D-glucose is sometimes referred to as dextrose, although the use of this name is strongly discouraged. The term dextrose is derived from *dextrorotatory glucose*.^[4] This name is therefore confusing when applied to the enantiomer, which rotates light in the opposite direction. Starch and cellulose are polymers derived from the dehydration of D-glucose. The other stereoisomer, called L-glucose, is hardly ever found in nature. The name "glucose" comes from the Greek word *glukus* meaning "sweet".

CHAPTER – 2

LITERATURE REVIEW

Oxalic acid is the simplest of dicarboxylic acid. Its name is derived from the Greek *Oxyes* meaning sharp, acidic referring to the acidity common in the foliage of certain plants from which it was first isolated. Oxalic acid is commercially available as the dihydrate containing 28.5% water. Anhydrous acid is odorless and white in colour. It is a monoclinic prism, particles size varying from fine powder to coarse granules which are also colorless, melting point 187°C of anhydrous form and 101.5°C of dihydrate form. Oxalic acid (HOOC-COOH), is one of the most widespread organic acid in plants. Oxalic acid is also produced in human organism as a product of metabolism, which is excreted with urine. Nevertheless, oxalic acid belongs to toxic ingredients of groceries. Binding metals causes symptoms of calcium deficiency. Its preparation from molasses is the most effective & cheapest way.

2.1 BRIEF FACT ABOUT MOLASSES^[1]

Molasses is a viscous by-product of the beating of sugarcane, grapes or sugarbeets into sugar. The word *molasses* comes from the Portuguese word *melaço*, which ultimately comes from *Mel*, the Latin word for "honey". The quality of Molasses depends on the maturity of the sugarcane or sugar beet, the amount of sugar extracted, and the method of extraction. Sweet sorghum is known in some parts of the United States as molasses. So molasses are of different kinds, based on their raw material. Sulfured molasses is made from young sugarcane. Sulfur dioxide, which acts as a preservative, is added during the sugar extraction process. Unsulphured molasses is made from mature sugarcane, which does not require such treatment. The three grades of molasses are: mild or Barbados, also known as first molasses; dark, or second molasses; and blackstrap. These grades may be sulphured or unsulphured. To make molasses, the cane of a sugar plant is harvested and stripped of its leaves. Its juice is extracted usually by crushing or mashing. The juice is boiled to concentrate it, which promotes the crystallization of the sugar. The result of this first

boiling and of the sugar crystals is first molasses, which has the highest sugar content because comparatively little sugar has been extracted from the source. Second molasses is created from a second boiling and sugar extraction, and has a slight bitter tinge to its taste. The third boiling of the sugar syrup yields blackstrap molasses, known for its robust flavour. The majority of sucrose from the original juice has been crystallised and removed. The food energy content of blackstrap molasses is still mostly from the small remaining sugar content. However, unlike refined sugars, it contains trace amounts of vitamins and significant amounts of several minerals. Blackstrap molasses is a source of calcium, magnesium, potassium and iron, one tablespoon provides up to 20% of the daily value of each of those nutrients.^[4] Blackstrap has long been sold as a health supplement. It is also used in the manufacture of ethyl alcohol for industry and as an ingredient in cattle feed. Molasses made from sugar beet is different from sugarcane molasses. Only the syrup left from the final crystallization stage is called molasses; intermediate syrups are referred to as high green and low green, and these are recycled within the crystallization plant to maximize extraction. Beet molasses is about 50% sugar by dry weight, predominantly sucrose, but also contains significant amounts of glucose and fructose. Beet molasses is limited in biotin (vitamin H or B₇) for cell growth; hence, it may need to be supplemented with a biotin source. The non sugar content includes many salts, such as calcium, potassium, oxalate, and chloride. It also contains the compounds Betaine and the Trisaccharide raffinose. These are either as a result of concentration from the original plant material or as a result of chemicals used in the processing, and make it unpalatable to humans. Hence it is mainly used as an additive to animal feed (called "molassed sugar beet feed") or as a fermentation feedstock. It is possible to extract additional sugar from beet molasses through a process known as molasses desugarisation.

This technique exploits industrial scale chromatography to separate sucrose from nonsugar

components. The technique is economically viable in trade-protected areas, where the price of sugar is supported above the world market price. Molasses is also used for production of Oxalic acid. Processes involved for production of oxalic acid from molasses are-

- 1) By using Nitric acid
- 2) By Fermentation process
- 3) By using *Aspergillus Niger*

2.2 PREPARATION OF OXALIC ACID BY MOLASSES BY USING NITRIC ACID^{[2],[5],[7]}

LABOTARY PREPARATION

The sugar beet molasses used in the experiment contained 48.1% sucrose, 3.2% invert sugar and 19.4% water. The chemicals employed were 65% HNO_3 and 93% H_2SO_4 . As catalyst, 7.5% V_2O_5 on porous silica gel support was used.

PROCESS IN DETAIL

Three reactor were used in the experiments. The details of the Experimental setup are given in Fig. 1. While the reactor 1 was used to produce oxalic acid from sugar beet molasses with mixed nitric acid and Sulphuric acid in the optimum conditions of it was simultaneously utilized as a source of nitrogen oxides needed as reactant in the second and third reactors. The reaction gases released from the final reactor were absorbed in the columns at 0°C . All of the reactants were prepared per 50 g molasses for each reactor. A predetermined amount of Vanadium Pentoxide catalyst was added to each reactor. The magnetic stirrers, heaters, cooling water and a cooler were turned on and air started to bubble through. Mixed acid was added to the reactor at a rate of 6 ml/min through a funnel to the neck of the ask. The $\text{HNO}_3 + \text{H}_2\text{SO}_4$ mixture was added from the dropping funnel as a thin stream. Initially the reaction in the reactor was started. The reaction temperature was controlled by adjusting the heating or cooling it with water through a water bath. The Oxides of Nitrogen evolving

from the reaction in the reactor were carried, by air to the second and third reactors. These Oxides were meant to act as oxidizers in the second and third reactors that contained enough H_2SO_4 , but no HNO_3 . In order to see how the conditions in the second reactor ejected the Oxalic acid yield, temperature, quantity of sulphuric acid, catalyst, additional water and total air flow rate for the two reactors were chosen as parameters. A similar study was carried out for the third reactor as well. After the addition of acid-mixture to the reactor, evolution of brown-coloured Nitrogen Oxides was visible immediately that indicates a rapid reaction. . However, it took the reaction in the second reactor 16 min to give visible gaseous products, and another 45 min period elapsed before any coloured gas evolution started in the third reactor.

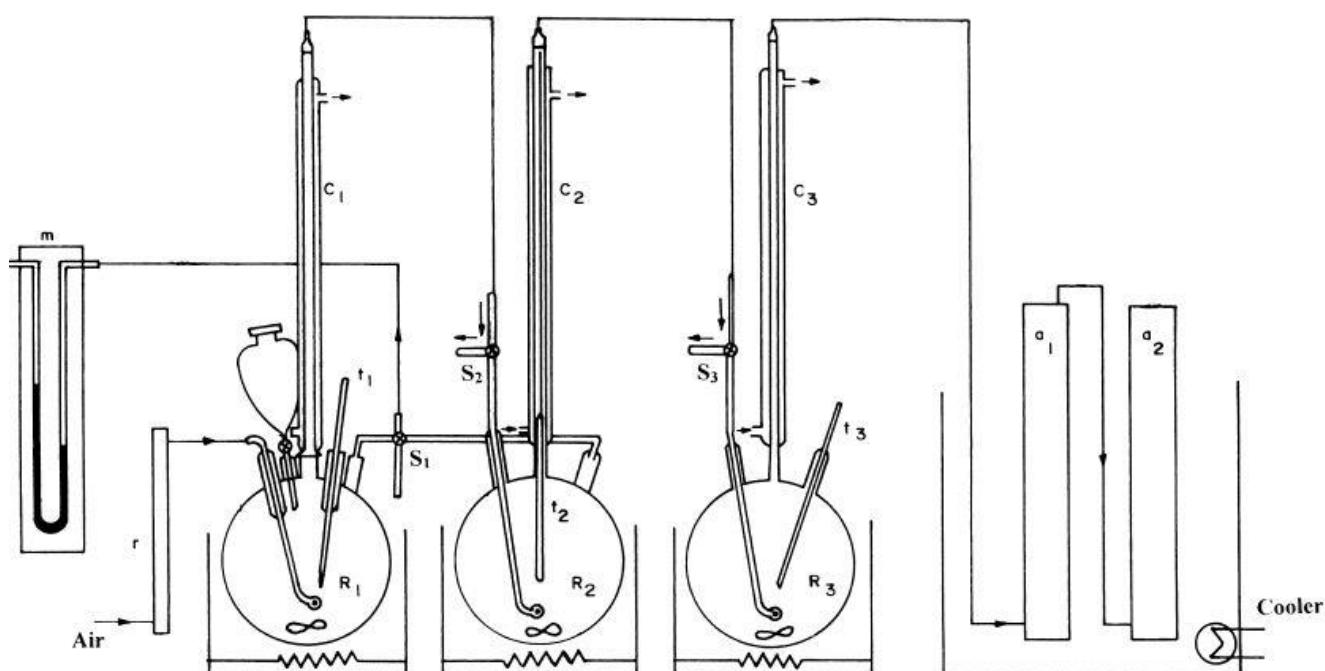


Fig. 1. Flow diagram of experimental setup; a1; a2: absorption column; C1;C2;C3: condenser; m: manometer; R1;R2;R3: reactor; r: rot meter; S1; S2; S3: sampling valve; t1; t2; t3: thermometer.

Based on these observations the delay for the start of the reaction in the third reactor was assumed to be 61 min and this was used in the kinetic studies. The reaction was assumed to be completed when the colour of the exhaust gases turned pale yellow. The reaction mixture was cooled to about 0°C and Oxalic acid crystals were separated by vacuum. Approximately eighty percent of the filtrate was evaporated gradually in a dish at about 45°C and then it was cooled to 5°C. The needle-shaped crystals of oxalic acid were separated again by vacuum filtration, and crystals were combined with the previous lot. For recrystallization, the crude product was dissolved in hot water, filtered and cooled to 0°C. Then, pure crystals were separated, and the residual filtrate was again evaporated in a dish at about 45°C by the same manner. The saturated solution was cooled to 0°C and filtered. The crystals were dried at about 60°C and weighed. The product was checked by determining the melting point, elemental composition and percent purity.

2.3 FERMENTATION BY USING POTTASIAM FERROCYANIDE^{[4],[6]}

Optimum conditions for treatment of this molasses sample, which in many respects is similar to the procedure of Eisenman, were found to be as follows: To 340 g of beet molasses partially diluted were added 0.60 gm of Potassium Ferrocyanide (in solution), and the whole was made to 1 liter with distilled water. Ten g of diatomaceous earth were added and mixed thoroughly. The medium thus prepared was allowed to stand overnight in a graduated cylinder or similar container at approximately 60°C. The medium was placed in the fermentation containers and autoclaved at 120°C for 15 minutes. Then it was kept still for 4 days. The final medium thus prepared contained approximately 15 per cent sugar. A total of four samples of straight house beet molasses from different localities were treated with Ferrocyanide. All of the samples gave satisfactory Oxalic acid & citric acid yields, although optimum conditions were established for only one sample, no. 1. The Ferrocyanide level, pH, and sterilization conditions required for maximum yields with the different samples was

varied considerably. Thus yields obtained with the four samples of molasses were sterilized with the pH unadjusted. Under these conditions, molasses no. 1 gave better results when sterilized, the other three apparently giving better results in an unsterile condition. No trouble was encountered from contamination of the unsterile media in the course of the fermentation, presumably because of the high concentration of acid formed. Further evidence of the necessity for determining the optimum treatment for a given lot of molasses in large-scale production became apparent in the case of molasses no. 2. The pH of this sample was 8.4, as compared to the nearly neutral reactions of the other samples. By adjusting the pH of this sample before Ferrocyanide treatment to an optimum, pH 6.0, and sterilizing the medium, total acidity yields as citric were increased from 90.5 to 91.5 per cent, and Oxalic acid yields, from 44.8 to 54.0 per cent.

2.4 BY USING ASPERGILLUS NIGER ^{[1],[11]}

Materials – Microorganism *Aspergillus Niger* NCIM 548 strain used in this study.

Medium composition- The medium contained the following components in (g/l): glucose (105.5) or sucrose (100) or lactose (102.6) plus glucose (2.51)—all with the same carbon content; NaNO₃ (1.5); KH₂PO₄ (0.5); MgSO₄*7H₂O (0.025); KCl (0.025); and yeast extract (1.6) Medium pH was adjusted to 6 with 4 M NaOH before sterilization. 20 ml of universal pH indicator solution was added per litre medium for observing its pH which was maintained within 6 to 7 by adding alkali during fermentation.

Spore suspensions -Spores from 7-day stationary culture at 30 °C in potato dextrose broth were suspended in 0.001% (v/v) Triton X-100 solution, and were counted under a light microscope in a Neubauer chamber.

METHOD:-

Fifty milliliter culture medium taken in a 250-ml Erlenmeyer flask was inoculated with spores of *Aspergillus Niger* (range: 5 to 6 per ml medium), and was incubated at 30 °C on an

orbital shaker at 215 rpm , & for 7 days it was kept in a dry condition . Culture filtrate from each triplicate set was analyzed for oxalic acid content. During fermentation, the pH of the medium was maintained at 6.5 with 4M NaOH. Growth and oxalic acid yield were monitored during the course of fermentation. Broths after fermentation were filtered through a 0.2-mm membrane. Oxalate in the filtrate was estimated by titration with KMnO_4 after precipitating with CaCl_2 . Sugars were estimated calorimetrically using Orcinol . Briefly, 1.5 ml of cold orcinol reagent (200 mg orcinol dissolved in 100 ml of 70% (v/v) H_2SO_4 in water) was added to 0.5 ml of diluted sample in a test tube, heated in a boiling water bath for 20 min, and then cooled. Biomass of *Aspergillus Niger* grown under different conditions was measured gravimetrically after separating the mycelia from fermented broth by filtration, washing with water, and drying at 80°C to constant weight.

2.5 COMPARATIVE STUDY

All of them uses raw material molasses for both the process (lab & industrial). For optimization , so we have to select the best process in term of cost & efficiency of products.

PROCESS	TIME REQUIRED	YIELD(%)
By using Nitric acid	2-3 hours	75
By using Potassium Ferrocyanide	3-4 days	52
By using <i>Aspergillus Niger</i>	7-8 days	61

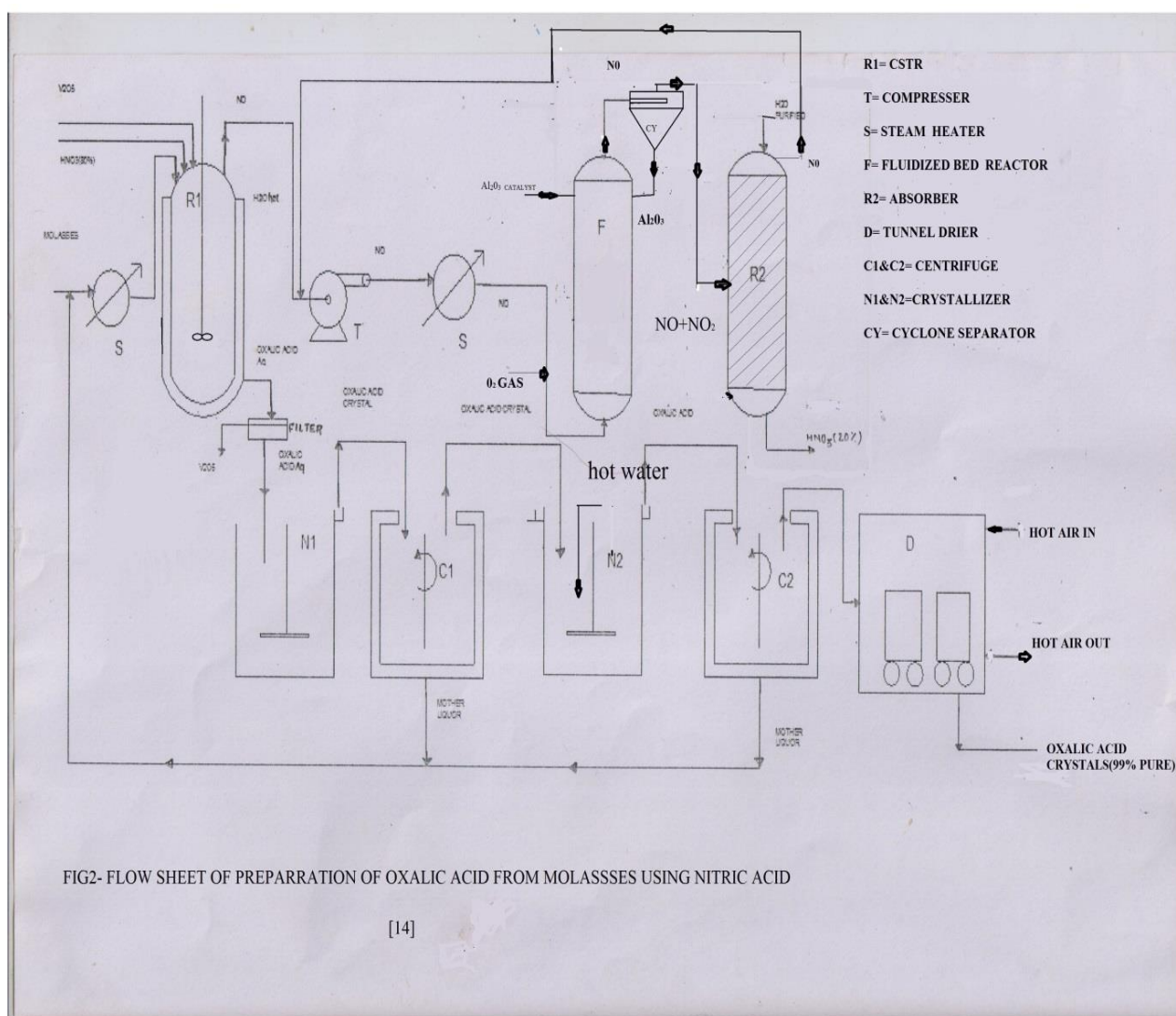
Table 1- Comparative analysis of all three process producing oxalic acid

2.6 BEST PROCESS ADOPTED

Except the first process all the process involved are fermentation process provide less yield and time taking , so preparation from nitric acid is adopted for the present work.

CHAPTER 3

PROCESS FLOW DIAGRAM & DESCRIPTION



Initially molasses was preheated & temperature of it was increased from 37°C to 65.5°C. After that it was fed into a CSTR. Simultaneously Nitric acid was also fed into the CSTR along with VanadiumPenta-oxide which act as a catalyst. This mixture was mixed thoroughly & was allowed to react for 2-3 hours. After 2-3 hours, Oxalic acid, un-reacted molasses, un-reacted Nitric acid, Nitrogen Oxide was formed. Oxalic acid along with un-reacted molasses, un-reactedNitric acid&VanadiumPenta-oxide comes out from the bottom section of CSTR and undergoes further separation process. During this process Vanadium Penta-oxide gets first get separated out with help of a filter . Oxalic acid & mother liquor(un-reacted Nitric acid & molasses) is separated in a 2 stage process. In the first stage , solution that has been filtered is fed into a crystallizer in which oxalic acid crystals along with mother liquor comes out and further these are separated with help of a centrifuge. After Oxalic acid gets separated , to remove inclusion (process by which a solvent particles get trapped inside a crystal) ,it is re-crystallized by adding hot water inside a crystallizer containing these Oxalic acid crystals. After separating out the mother liquor again , Oxalic acid crystals are sent into drier to remove the moisture present on the surface of the crystals. Nitrogen Oxide gas which comes out from the top surface of CSTR cannot be discharged directly to the atmosphere, as it can cause air pollution, so this gas was sent into a compressor to increase the pressure & to a steam heater to increase the temperature. After that this gas was sent to fluidized-bed reactor & in presence of Al_2O_3 catalyst, Nitrogen dioxide gas is formed. Al_2O_3 after this get separated by cyclone separator. After this process, in an absorber, Nitrogen dioxide gas was allowed to react with water sprayed inside the absorber to produce Nitric acid & Nitrogen oxide gas . Nitric acid (20%)obtained which is less concentrated than the Nitric acid (95%)which was used as a feed can further be used in other industrial process and Nitrogen oxide gas can be recycled back to stream leading to fluidized bed reactor.

CHAPTER -4

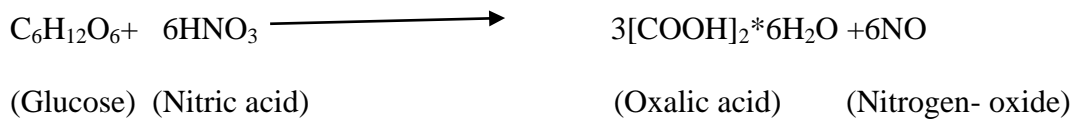
MASS BALANCE

It is desired to produce 30 tons per day of Oxalic acid using molasses as the starting material. Accordingly the material balance is presented below

4.1 MATERIAL BALANCE FOR THE CSTR

Oxalic acid to be produced= 30000kg
=333.333kmol

Reaction-



1kmol of Glucose gives 3kmol of oxalic acid

Therefore 1 kmol of Oxalic acid is produced from 0.333 kmol of glucose

Therefore 333.3 kmol of Oxalic acid is produced from = $333.333/3=111.111$ kmol of glucose

Yield is 75% (assumed)

Therefore Glucose actually taken= $111.1111/0.75=148.156$ kmol

Therefore Glucose un-reacted= $148.156-111.111=37.045$ kmol

1 kmol of Glucose reacts with 6 kmol of Nitric acid

Therefore 148.156 kmol of Glucose reacts with $148.156*6=888.936$ kmol of Nitric acid

Yield is 75% (assumed)

So Nitric acid reacted = $888.9*0.75=666.67$ kmol

Un-reacted Nitric acid= $888.9-666.67=222.22$ kmol

Water produced= 6kmol per 1kmol of Glucose reacted

So total water produced due to reaction of $111.1*6=666.66$ kmol

Again Nitrogen oxide produce= 666.77 kmol

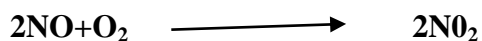
Vanadium Penta-oxide used = 0.2% of feed = 29.65 kmol

INPUT			OUTPUT		
COMPONENT	FEED IN KMOL	FEED IN KG	COMPONENT	IN KMOL	IN KG
Glucose	148.15	26667.1	Glucose (un-reacted)	37.04	6667.2
Nitric acid	666.67	56000.7	Nitric acid (un-reacted)	222.2	14000.1
Vanadium Penta-oxide	29.65	5337	Water	666.6	119998.8
			Oxalic acid	333.3	30000
			Nitric oxide	666.6	20000.1
			Vanadium Penta-oxide	29.65	5337
TOTAL		88004.8	TOTAL		88004.8

Table 2– mass balance for the CSTR

4.2 MATERIAL BALANCE FOR THE FLUIDIZED BED REACTOR

REACTION INVOLVED



666.6kmol of NO gas enters into the reactor

Coverion of NO gas toNO₂is 90% (assumed)

So amount of NO reacted=666.6*0.9=599.94 kmol

Un-reacted NO=66.6 kmol

So from the reaction

1kmol of NO reacts with = 0.5 kmol of Oxygen

So 599.94 kmol of NO reacts with= 599.94*0.5=299.98=300kmol of Oxygen

Considering total consumption of Oxygen gas

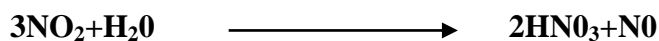
Amount of NO₂produced from reaction is=599.94kmol

Al₂O₃ used = 0.5% of feed= 3.33kmol

INPUT			OUTPUT		
COMPONENT	FEED IN KMOL	FEED IN KG	COMPONENT	FEED IN KMOL	FEED IN KG
NO	666.6	19998.01	NO ₂	599.94	27597.24
O ₂	300	9600	Un-reacted NO	66.66	1999.8
Al ₂ O ₃	3.33	339.66	Al ₂ O ₃	3.33	339.66
TOTAL		29937.67	TOTAL		29937.67

TABLE 3- Mass balance for the FBR

4.3 MATERIAL BALANCE FOR THE ABSORBER



So from the reaction from 1 kmol of Nitrogen di - oxide gas = 0.66kmol of Nitric acid is produced.

Let the conversion be 80% (assumed)

Amount of NO₂ entering in to the absorber=599.94 kmol.

So amount of NO₂ gas reacted $0.8 \times 599.94 = 479.95$ kmol

So 479.955 kmol of NO₂ on conversion will give $0.66 \times 479.95 = 319.97$ kmol of Nitric acid

Un-reacted NO₂ gas = $599.94 - 479.95 = 119.99$ kmol

From 1 kmol of NO₂ = 0.3 kmol of Water is required

For 479.95 kmol of Nitrogen dioxide = $479.95 \times 0.3 = 143.98$ kmol of Water is required

From 1 kmol of NO₂ = 0.33 kmol of NO gas is produced

So for 479.95 kmol of NO₂ = $0.33 \times 479.95 = 159.98$ kmol of NO gas is produced

Un-reacted NO gas in input = 66.66 kmol

Amount of NO gas produced = 160 kmol

Total NO gas output = $66.66 + 160 = 226.66$ kmol

INPUT			OUTPUT		
COMPONENT	FEED IN KMOL	FEED IN KG	COMPONENT	FEED IN KMOL	FEED IN KG
NO ₂	599.94	26397.36	NITRIC ACID	319.97	20158.11
H ₂ O	143.98	2588.4	NO	226.6	6798
Un-reacted NO ₂ gas	66.66	1999.9	un-reacted NO ₂ gas	120	5280
TOTAL		30985.56			30985.56

Table 4- mass balance for the Absorber

4.4 MATERIAL BALANCE FOR THE CRYSTALLIZER 1

Initial temperature of Oxalic acid + un-reacted mixture = 65.5°C

Final temperature of Oxalic acid + un-reacted mixture = 25°C

Mass of Oxalic acid + un-reacted mixture = 62667.1 kg

Solubility of Oxalic acid at 65.5°C = 80.5 mol/100 mol of Water

Solubility of Oxalic acid at 25°C = 46.6/100 mol of Water

$$x_f = 80.5/180.5 = 0.445$$

$$x_c = 1$$

$$x_m = 46.6/146.6 = 0.318$$

$F = C + M$ (for liquid phase reaction)

$$C + M = F$$

$$62667.1 = C + M \dots\dots\dots (1)$$

We know $F \cdot x_f = C \cdot x_c + M \cdot x_m$

Where F = feed

C = weight of crystal

M = weight of mother liquor

$$\Rightarrow 62667.1 \times 0.445 = C \times 1 + 0.318 \times M \dots\dots\dots(2)$$

Upon solving (1) & (2) we get C= 30303.1 kg & M= 32364.1kg

4.5 MATERIAAL BALANCE FOR THE CRYSTALLIZER 2

Initial temperature of Oxalic acid = 90°C

Final temperature of Oxalic acid = 25°C (on cooling)

Solubility of Oxalic acid at 90°C = 112.6mol/100 mol of Water

Solubility of Oxalic acid at 25°C= 46.6/100mol of Water

$$X_f = 112.6 / 212.6 = 0.529$$

$$X_c = 1$$

$$X_m = 46.6 / 146.6 = 0.318$$

$$F = C + M \text{ (for liquid phase reaction)}$$

$$C + M = F$$

$$30303.1 = C + M \dots\dots\dots(1)$$

$$\text{We know } F \times X_f = C \times X_c + M \times X_m$$

Where F=feed

C= weight of crystal

M= weight of mother liquor

$$\Rightarrow 30303.1 \times 0.529 = C \times 1 + 0.318 \times M \dots\dots\dots(2)$$

Upon solving (1) & (2) we get C= 30819.1kg & M= 221.1kg

4.6 MATERIAAL BALANCE FOR THE CENTRIFUGE 1

Amount of feed entering to the centrifuge=62667.1kg

After separation amount of oxalic acid crystals obtained=30303.1kg

Amount of mother liquor obtained=32364kg

4.7 MATERIAAL BALANCE FOR THE CENTRIFUGE 2

Amount of feed entering to the centrifuge-30303.1kg

After separation amount of oxalic acid crystals obtained=30819.1 kg

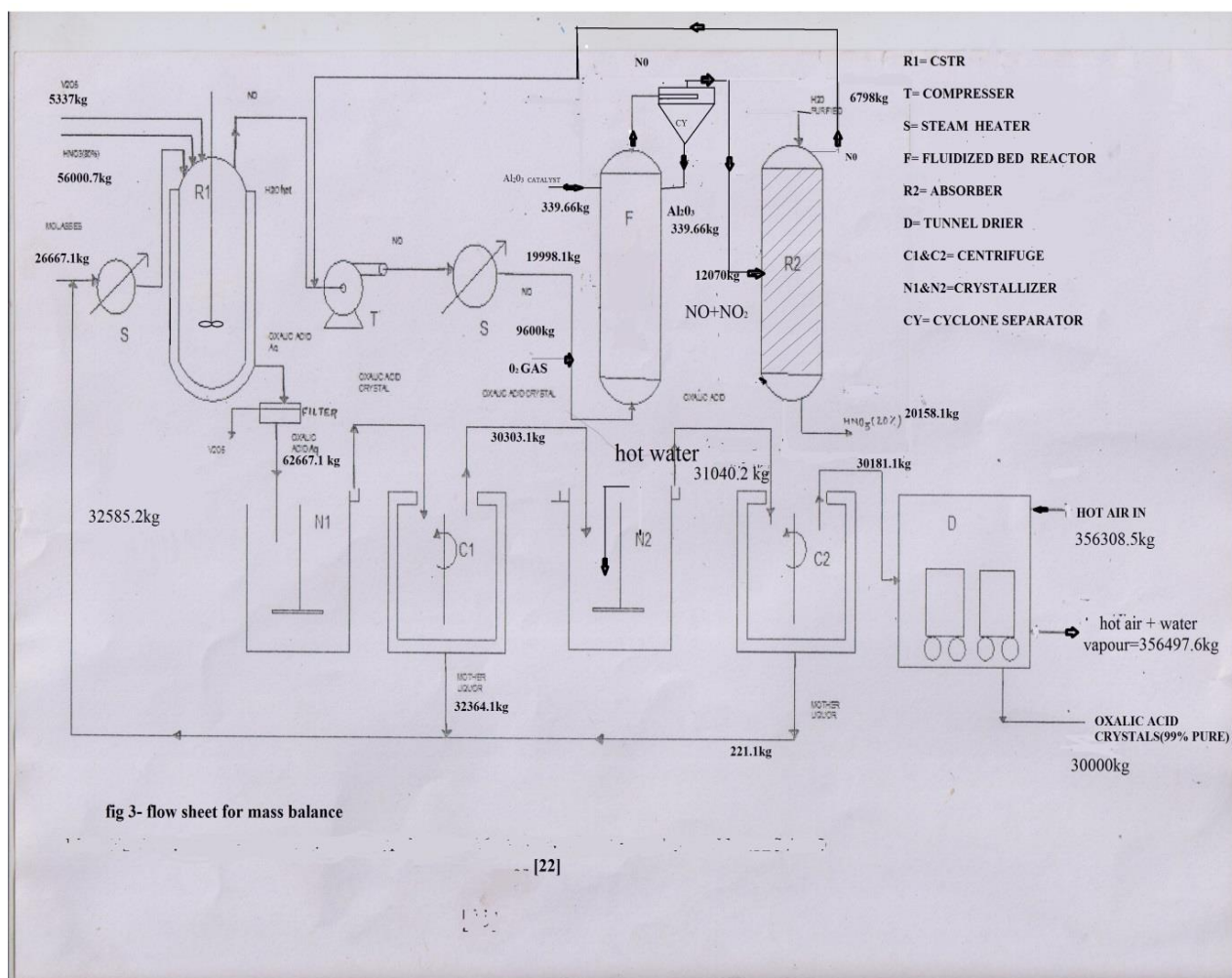
Amount of mother liquor obtained=221.1 kg

4.8 MATERIAL BALANCE FOR THE DRIER

For evaporation of 1 kg of water at 1 atm of pressure ,hot air requirement is $10-20 \text{ m}^3 = 15^*$

$29 = 435 \text{ kg}$

So for 819.1 kg , amount of air needed $435 \times 819.1 = 356308.5 \text{ kg}$



CHAPTER -5

ENERGY BALANCE

5.1 ENERGY BALANCE FOR THE CSTR

REACTION INVOLVED



TOTAL ENTHALPHY OF THE REACTANT

We know $H(\text{enthalpy}) = mc_p \Delta t$

$$H_{\text{glucose}} = 26667.2 * (1.21/1000) * 65.5 = 211350.1 \text{ kJ/kg}$$

$$H_{\text{Nitric acid}} = 56000.78 * (1.74/1000) * 65.5 = 6382.32 \text{ kJ/kg}$$

$$H_{\text{reactant}} = H_1 = 217732.421 \text{ kJ}$$

TOTAL ENTHALPHY OF THE PRODUCT

$$H_{\text{Oxalic acid}} = 30000 * (1.2/1000) * 65.5 = 2358 \text{ kJ/kg}$$

$$H_{\text{Nitric oxide}} = 20000.1 * (1.114/1000) * 65.5 = 1454.1 \text{ kJ/kg}$$

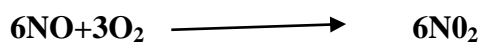
$$H_{\text{product}} = H_2 = 3812.1 \text{ kJ/kg}$$

$$Q(\text{heat}) = H_{\text{product}} - H_{\text{reactant}} = H_2 - H_1 = 3812.1 - 217732.42 = -213920.321 \text{ kJ (exothermic)}$$

Here specific heat " c_p " value was obtained through sources from internet

5.2 ENERGY BALANCE FOR THE FLUIDIZED BED REACTOR

REACTION INVOLVED



TOTAL ENTHALPHY FOR THE REACTANT

We know $H(\text{enthalpy}) = mc_p \Delta t$

$$H_{\text{NO}} = (1.394/1000) * 19998.01 * 85.5 = 2369.564 \text{ kJ/kg}$$

$$H_{\text{O}_2} = 0.934 * 5332.8 * 85.5 = 423.37 \text{ kJ/kg}$$

$$\text{So } H_1 = H_{\text{NO}} + H_{\text{O}_2} = 2369.564 + 423.37 = 2786.9 \text{ kJ/kg}$$

For a fluidized bed reactor, temperature of Nitrogen oxide gas temperature was raised to 85.5°C

TOTAL ENTHALPHY FOR THE PRODUCT

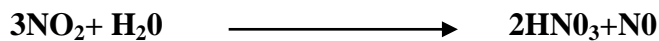
$$H_{\text{NO}_2} = 1.431 * 25330.6 * 85.5 = 3081087.53 \text{ kJ/kg}$$

$$\text{So } H_2 = 3081.087 \text{ kJ/kg}$$

$$Q(\text{heat}) = H_2 - H_1 = 3081.087 - 2786.9 = 294.152 \text{ kJ}$$

5.3 ENERGY BALANCE FOR THE ABSORBER

REACTION INVOLVED



We know $H(\text{enthalpy}) = mc_p \Delta t$

TOTAL ENTHALPHY FOR THE REACTANT

$$H_{\text{NO}_2} = 25330.6 * (1.431/1000) * 85.5 = 3081.087 \text{ kJ/kg}$$

$$H_{\text{H}_2\text{O}} = 3999.6 * 4.18 * 85.5 = 1421.05 \text{ kJ/kg}$$

$$H_1 = 3081.087 + 1421.057 \text{ KJ} = 4502.145 \text{ kJ/kg}$$

TOTAL ENTHALPHY FOR THE PRODUCT

$$H_{\text{NO}} = 6666.1 * (1.31/1000) * 85.5 = 759.268 \text{ kJ/kg}$$

$$H_{\text{HNO}_3} = 0.018 * 85.5 * 25197.48 = 3876.63 \text{ kJ/kg}$$

$$Q = (\text{heat}) H_2 - H_1 = 4635.901 - 4502.14 = 4233.755 \text{ kJ}$$

5.4 ENERGY BALANCE FOR THE STEAM HEATER 1

Initial temperature of molasses = 37°C

Final temperature of molasses = 65.5°C

$$Q = mc_p \Delta t = 26667.2 * 0.0121 * (65.5 - 37) = 92888.888 \text{ kJ}$$

so amount amount of Water required in steam heater = $Q / \text{latent heat of vaporization}$ so we

know latent heat of vapourization = 2270

$$92888.88 / 2270 = 409.2 \text{ kg of Water.}$$

So 409.2 kg water is required in the steam heater

5.5 ENERGY BALANCE FOR THE STEAM HEATER 2

Initial temperature of NO Gas =65.5°C

final temperature NO gas =85.5°C

$$Q = m c_p \Delta t = 19998.01 * 0.014 * 20 = 439956.22 \text{ kJ}$$

So amount of Water required for steam heater = $439956.22 / 2270 = 193.81 \text{ kg}$ of Water

5.6 ENERGY BALANCE FOR THE CRYSTALLIZER 1

HEAT INPUT-

$$\text{With Water} = x * 4.18 * 65.5$$

$$\text{With Oxalic acid} = 30000 * 65.58 * 1.238$$

HEAT OUTPUT-

$$\text{With Water} = x * 4.18 * 65.5$$

$$\text{With Oxalic acid crystals} = 30303.1 * 1.238 * 37$$

As Heat input = Heat output

$$x * 4.18 * 65.5 + 30000 * 65.5 * 1.328 = 30303.1 * 1.23 * 37 + x * 4.18 * 65.5$$

Upon calculation $x = 9189.11 \text{ kg}$ was found out

5.7 ENERGY BALANCE FOR THE CRYSTALLIZER 2

HEAT INPUT-

$$\text{With Water} = x * 4.18 * 80$$

$$\text{With Oxalic acid} = 30303.1 * 80 * 1.238$$

HEAT OUTPUT-

$$\text{With Water} = x * 4.18 * 25$$

$$\text{With Oxalic acid crystals} = 30000.12 * 1.238 * 25$$

As Heat input = heat output

$$x * 4.18 * 65.5 + 30303.1 * 80 * 1.328 = 30000.12 * 1.23 * 37 + x * 4.18 * 65.5$$

Upon calculation $x = 7250.911 \text{ kg}$ was found out

5.8 ENERGY BALANCE FOR THE DRIER

Amount of Oxalic acid crystal required=30000 kg

Amount of Oxalic acid fed into the drier=30819.1kg

Therefore amount of water evaporated = 30819.1-30000 =819.1kg

Inlet temperature of feed= 25°C

required temperature of hot air = t°C

$$\Delta t = t^{\circ}\text{C} - 25^{\circ}\text{C}$$

Latent heat of vapourization= 2270 kJ/kg

Therefore heat required to evaporate 819.1 kg of water=q=2270*819.1=186140 kJ

Specific heat of water= 4.187 kJ/kg °C

Mass of water =819.1 kg

Given temperature $\Delta t = t^{\circ}\text{C} - 25^{\circ}\text{C}$

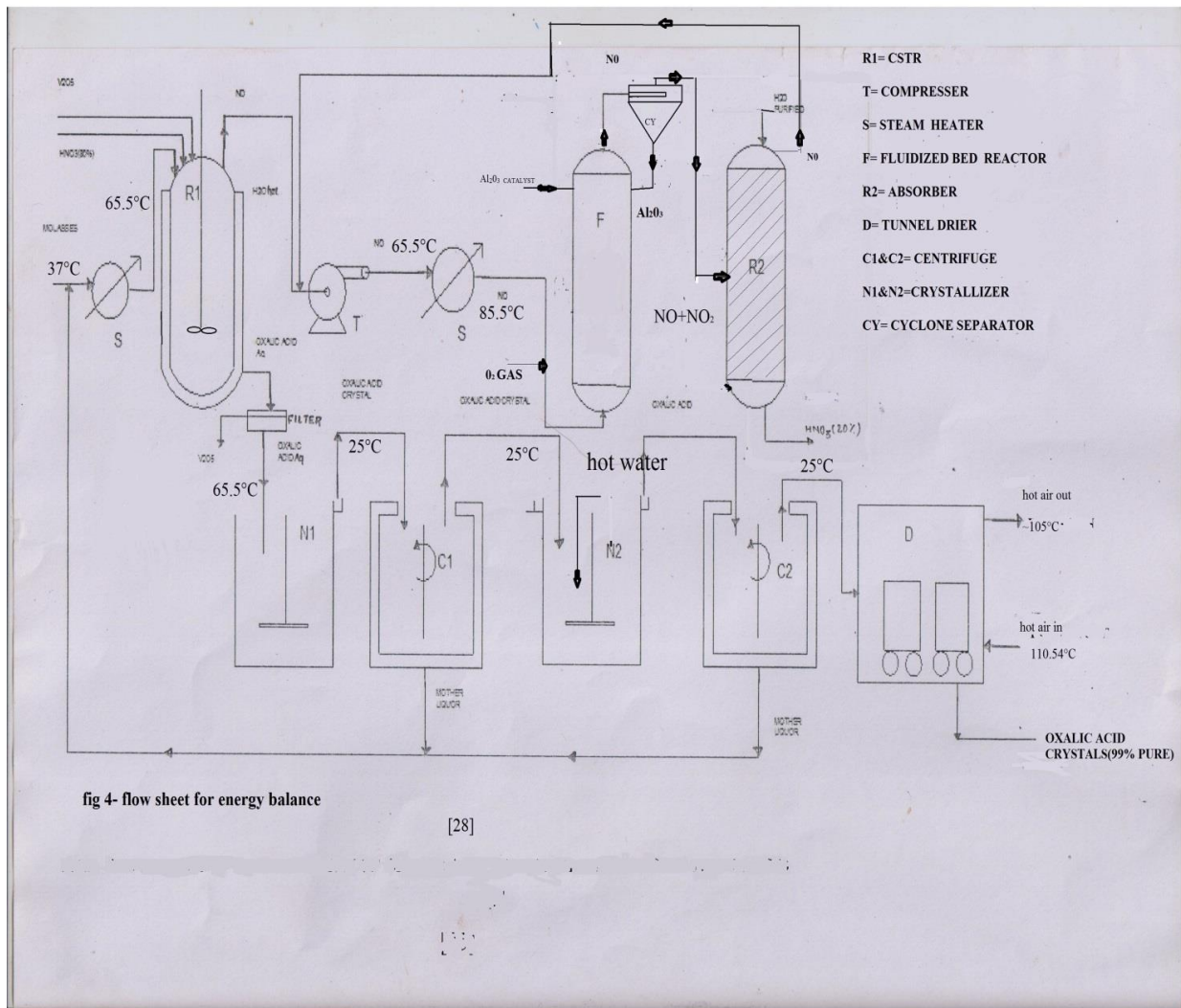
But according to energy balance

$$mc_p\Delta t = m\lambda$$

$$\Rightarrow 819.1 * 4.187 * (t^{\circ}\text{C} - 25^{\circ}\text{C}) = 186140$$

$$\Rightarrow t^{\circ}\text{C} = 85.54 + 25 = 110.54^{\circ}\text{C}$$

No energy balance is required for centrifuge as inlet & outlet temperature is same.



[28]

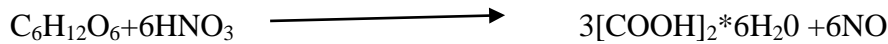
CHAPTER 6

MECHANICAL & PROCESS DESIGN

DESIGN OF THE CSTR

$$V/F_{A0} = \tau/C_{A0} = (X_A/-r_A) \quad \text{- Formula to be used}$$

Where C_{A0} = molar flow rate



Above reaction is 1st order reaction

Where rate of reaction is directly proportional to initial concentration of reactant

$$\text{So } dr_a = k_1[\text{Oxalic acid}]$$

Feed entering : Glucose = 148.15 kmol = 26667 kg & Nitric acid = 888.9 kmol = 56000 kg

Density of Glucose = 1567.7 kg/m³

Density of Nitric acid = 1420 kg/m³

Therefore v_1 = volumetric flow rate = $(26667/1567.7 + 56000/1420) \text{ m}^3/\text{day} = 57.12 \text{ m}^3/\text{day}$

Concentration = molar.flow.rate/volumetric.flow.rate

$$(\text{kmol/day})/(\text{m}^3/\text{day}) = 1037.05/57.12 = 18.16 \text{ m}^3$$

$$X_a = 1/7 = 0.14$$

$$C_A/C_{A0} = 1 - x_a/1 + x_a = 1 - 0.14/1 + 0.14 = 0.75$$

$$\text{So } C_A = 13.62 \text{ kmol/m}^3$$

Rate constant calculation

$$K = -1/a * d[a]/dt = -1/a * (C_{A0} - C_A)$$

$$= 0.3 * 4.54 \text{ day}^{-1}$$

$$= 1.52 \text{ day}^{-1}$$

$$\tau = (C_{A0} - C_A) * X_a / k * C_A$$

$$4.54 * 0.14 / 1.52 * 13.62$$

$$0.03 \text{ day} (43.2 \text{ min})$$

$$V_2 = \tau * v_1 = 0.03 * 57.12 = 1.72 \text{ m}^3$$

Diameter & length calculation

$$V_2 = \pi/4 * d^2 * l$$

For CSTR l/d ratio =3

$$l = 3 * d$$

$$\text{So } d^3 = 1.72 * 4 / \pi * 3$$

$$D = 0.9\text{m}$$

$$L = 2.7\text{m}$$

MECHANICAL DESIGN (CSTR)

DESIGN & SUPPORT

$$D = 0.9\text{m}$$

$$L = \text{height} = 2.7\text{m}$$

Assuming appropriate weight of vessel = weight of vessel + weight of

$$\text{accessories} = 1500000\text{kg} = w_{\text{max}}$$

$$\text{wind pressure} = 130\text{kg/m}^3$$

stress due to dead weight (ref fig 13.19 , MV JOSHI)^[18]

$$f_d = w(\pi * d * t_{sk}) = 1500000 * \pi * 0.9 * t_{sk} = 530516.47 / t_{sk} \text{ kg/m}^2$$

STRESS DUE TO WIND LOAD

For vessel less than 20m height

$$m_w = p_{bw} / (h/2)$$

$$k_1 = 0.7 \text{ for cylindrical surface}$$

$$k_2 = 1.0 \text{ if the period of vibration is less than } 0.5$$

So maximum stress at bottom

$$f_{1\text{max}} = (f_{wb} + f_{sb}) - f_{db}$$

$$= (469.21 + 190985.93) - 530516.47 = 339061.26\text{kg/m}^2$$

Permissible tensile stress for structure steel

$$F = 1400000 \text{ kg/m}^3$$

$$T_{sk} = 339061.26 / 1400000 = 0.2 \text{ m}$$

SKIRT BEARING PLATE

$$\sigma_c = w_1/a + m_w/z$$

$$W_1 = \text{weight of plate} = 1500000 * 9.81 = 14715 \text{ kN}$$

$$A = \pi * 0.1 * 2.7 = 0.84 \text{ m}^2$$

$$P_{bw} = k_1 * k_2 * p_1 * h_1 * d_0$$

$$= 0.7 * 1 * 0.9 * 2.7 * 130 = 221.13 \text{ kg/m}$$

$$M_w = 221.13 * (2.7/2) = 298.5 \text{ kgm}$$

$$F_{wb} = (4 * m_w) / \pi * d_0^2 * t_{sk}$$

$$= 4 * 298.5 / \pi * 0.81 * t_{sk} = 469.21 \text{ kg/m}^2$$

From equation 13,27 in M.V. Joshi ^[18]

$$f_{sb} = 2/3 (c * w * h) / \pi * d * d_{tsk}$$

$$C = 0.2 \text{ assumed}$$

$$f_{sb} = 0.6 (0.2 * 1500000 * 2.7 / \pi * 0.9 * 0.9 * t_{sk}) = 190985.93 \text{ kh/m}^2$$

Z = compressibility

$$Z = \pi * 0.45 * 0.45 * 2.7 = 1.71 \text{ m}^3$$

$$\sigma_m = \text{maximum compressibility} = 147.15 / 1.33 + 575.316 / 1.71 = 1105408.4 \text{ N/m}^2$$

It should be in range of 5.2 to 12.5 so above factor is satisfactory

$$\text{So } t_{bp} = 2.7 (3 * 11.06 * 50)^{0.5}$$

Anchor bolts

Assuming minimum weight of vessel = 15000 kg

From eqn. 13.33 of MV JOSHI ^[18]

$$\sigma_m = (W_{min}/a) - (m_w/z) = (15000/0.84) - (298.5/1.71) = 17682.5 \text{ N/m}^2$$

From eqn. 10.12 of B.C.B. ^[19]

$$R_{IN}=0.55 \text{ m}$$

$$\text{So } j = W_{\min} * R_{IN}/mw = 15000 * 0.55 / 298.5 = 27.6 \text{ m}$$

As $j > 1.5$, the vessel is steady by its own weight, so anchor bolt is not required

DESIGN OF COOLING WATER JACKET

Considering its height = $0.9 * \text{height of CSTR} = h_j = 2.43 \text{ m}$

Heat evolved in CSTR = 213920.32 kJ

We know $Q(\text{heat}) = m C_p \Delta t$

$$\Rightarrow m * 4.2 * (96 - 30) = 213920.32 \text{ KJ}$$

$$\Rightarrow m = (213920.32) / (4.2 * 66) = 771.7 \text{ kg of water/day}$$

Now for Density of water = 1000 kg/m^3

So volume of water needed = $0.77 \text{ m}^3/\text{day}$

As the jacket surrounds the CSTR. So, it is assumed as hollow cylinder

$$\text{So } \pi * h * (R_2^2 - R_1^2) = 0.77 \text{ m}^3$$

R_1 is radius of CSTR = 0.45 m

$$R_2^2 - 0.2025 = 0.77 / (\pi * 2.43)$$

$$R_2 = 0.56 \text{ m}$$

So annular thickness of jacket = $R_2 - R_1$

$$= 0.56 - 0.45$$

$$= 0.09 \text{ m}$$

Or, 9 cm

(which is a permeable thickness of jacket)

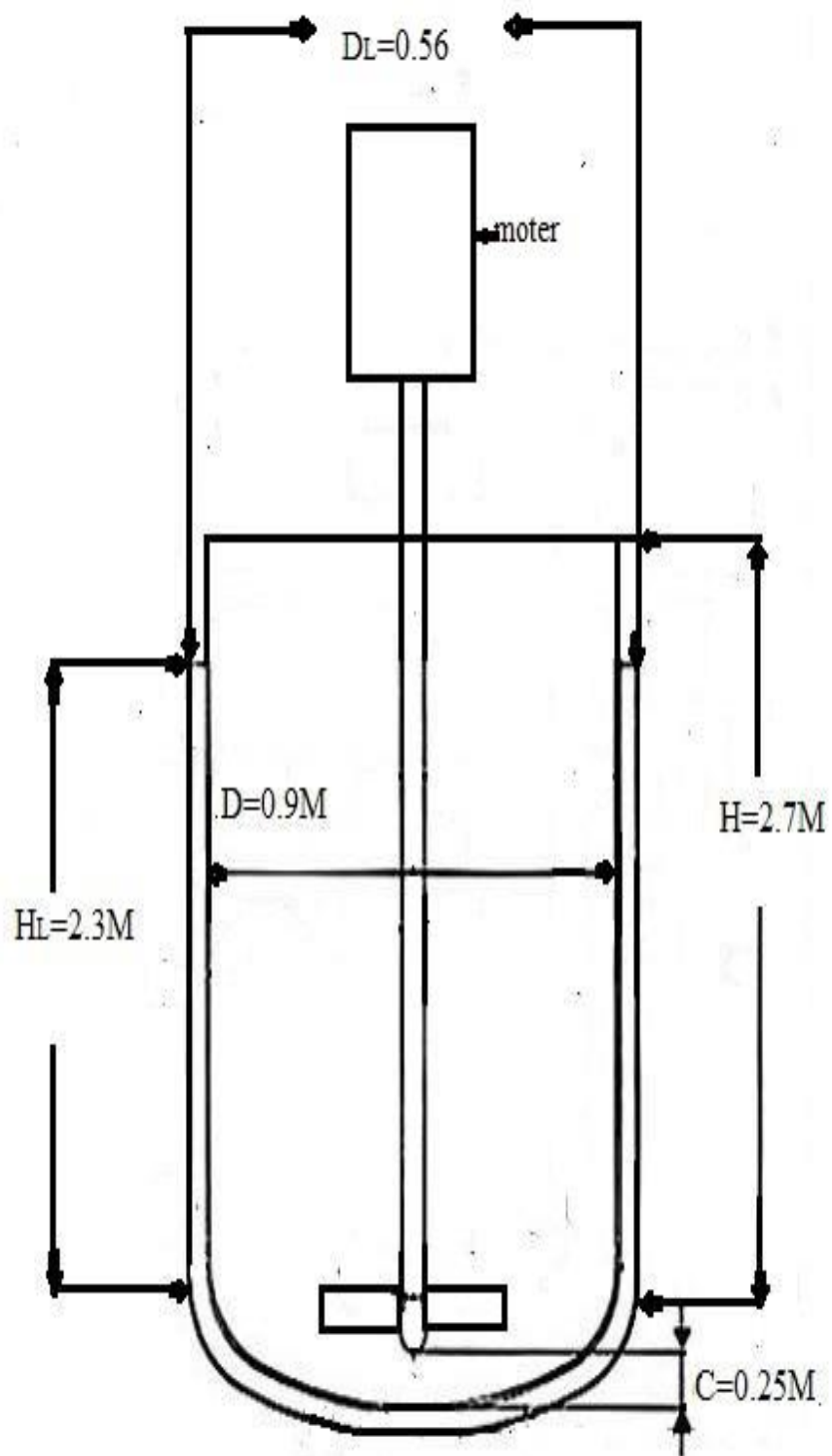


Fig 5- design of continuous stirred tank reactor

DESIGN OF THE CRYSTALLIZER

Let v_1 = volume of crystallizer

Sp_1 = (specific gravity of solution entering to the crystallizer)

Now for a crystallizer

$Sp_1 = \frac{\text{total mass of soln.}(m_1) * \text{total mass of the component that need to be crystallized}(m_2)}{E(m_2) + E(m_1)}$

Where E = efficiency of crystallizer

Considering $E = 80\%$

$Sp_1 = \frac{62667.1 * 30000}{(0.8 * 30000) + (0.2 * 62667.1)} = 51460.08$ (unitless)

Quality = mass of crystal obtained / (100 - E) = $\frac{30303.1}{0.2} = 151515 \text{ m}^3$

So volume $v_1 = \frac{\text{quality}}{6.24(sp_1)} = \frac{151515.5}{6.24 * 51460.08} = 0.471 \text{ m}^3$

CALCULATION OF LENGTH & DIAMETER

$V_1 = \frac{\pi * D^2 * H}{4}$

Where D = diameter & H = height

$0.471 = \frac{\pi * D^2 * H}{4}$ -(1)

For a crystallizer = $H = 2.5D$

$0.471 = \frac{\pi * 2.5 * H * H}{4}$

$\Rightarrow H = 4.1 \text{ m}$

So $D = \frac{2.5}{4.1} = 0.6 \text{ m}$

MECHANICAL DESIGN & SUPPORT

$D = 0.6 \text{ m}$

$L = \text{height} = 4.1 \text{ m}$

Assuming appropriate weight of vessel = weight of vessel + weight of

accessories = $1500000 \text{ kg} = W_{\text{max}}$

wind pressure = 130 kg/m^3

stress due to dead weight (ref fig 13.19 , MV JOSHI)^[18]

$$f_d = w(\pi * d * t_{sk}) = 1500000 * \pi * 0.6 * t_{sk} = 420516.47 / t_{sk} \text{ kg/m}^2$$

STRESS DUE TO WIND LOAD

for vessel less than 20m height

$$m_w = p_b w / (h/2)$$

$k_1 = 0.7$ for cylindrical surface

$k_2 = 1.0$ if the period of vibration is less than 0.5

so maximum stress at bottom

$$f_{1\max} = (f_{wb} + f_{sb}) - f_{db}$$

$$= (469.21 + 190985.93) - 530516.47 = 339061.26 \text{ kg/m}^2$$

Permissible tensile stress for structure steel

$$F = 1400000 \text{ kg/m}^2$$

$$T_{sk} = 339061.26 / 1400000 = 0.24 \text{ m}$$

SKIRT BEARING PLATE

$$\sigma_c = W_1 / a + m_w / z$$

$$W_1 = \text{weight of plate} = 1500000 * 9.81 = 14715 \text{ kN}$$

$$A = \pi * 0.1 * 2.7 = 0.84 \text{ m}^2$$

$$P_{bw} = k_1 * k_2 * p_1 * h_1 * d_0$$

$$= 0.7 * 1 * 0.9 * 4.2 * 130 = 621.13 \text{ kg/m}$$

$$M_w = 221.13 * (2.7/2) = 298.5 \text{ kgm}$$

$$F_{wb} = (4 * m_w) / (\pi * d_0^2 * t_{sk})$$

$$= 4 * 298.5 / (\pi * 0.81 * t_{sk}) = 469.21 \text{ kg/m}^2$$

From equation 13,27 in M.V. Joshi ^[18]

$$F_{sb} = 2/3 (c * w * h) / (\pi * d * d * t_{sk})$$

$C = 0.2$ assumed

$$F_{sb}=0.6(0.2*1500000*2.7/\pi*0.9*0.9*t_{sk})=190985.93\text{kh/m}^2$$

Z= compressibility

$$Z=\pi*0.45*0.45*2.7=1.71\text{m}^3$$

$$\sigma_m=\text{maximum compressibility}=147.15/1.33+575.316/1.71=905408.4\text{ N/m}^2$$

It should be in range of 5.2 to 12.5 so above factor is satisfactory

$$\text{So } t_{bp}=2.7(3*11.06*150)^{0.5}$$

Anchor bolts

Assuming minimum weight of vessel= 15000kg

From eqn. 13.33 of MV JOSHI

$$\sigma_m=(W_{in}/a)-(mw/z)=(15000/0.84)-(298.5/1.71)=9682.5\text{N/m}^2$$

From eqn. 10.12 of B.C.B.^[19]

$$R_{IN}=0.34\text{ m}$$

$$\text{So } j=W_{min}*R_{IN}/mw=15000*0.55/298.5=16.6\text{m}$$

As $j>1.5$, the vessel is steady by its own weight , so anchor bolt is not required

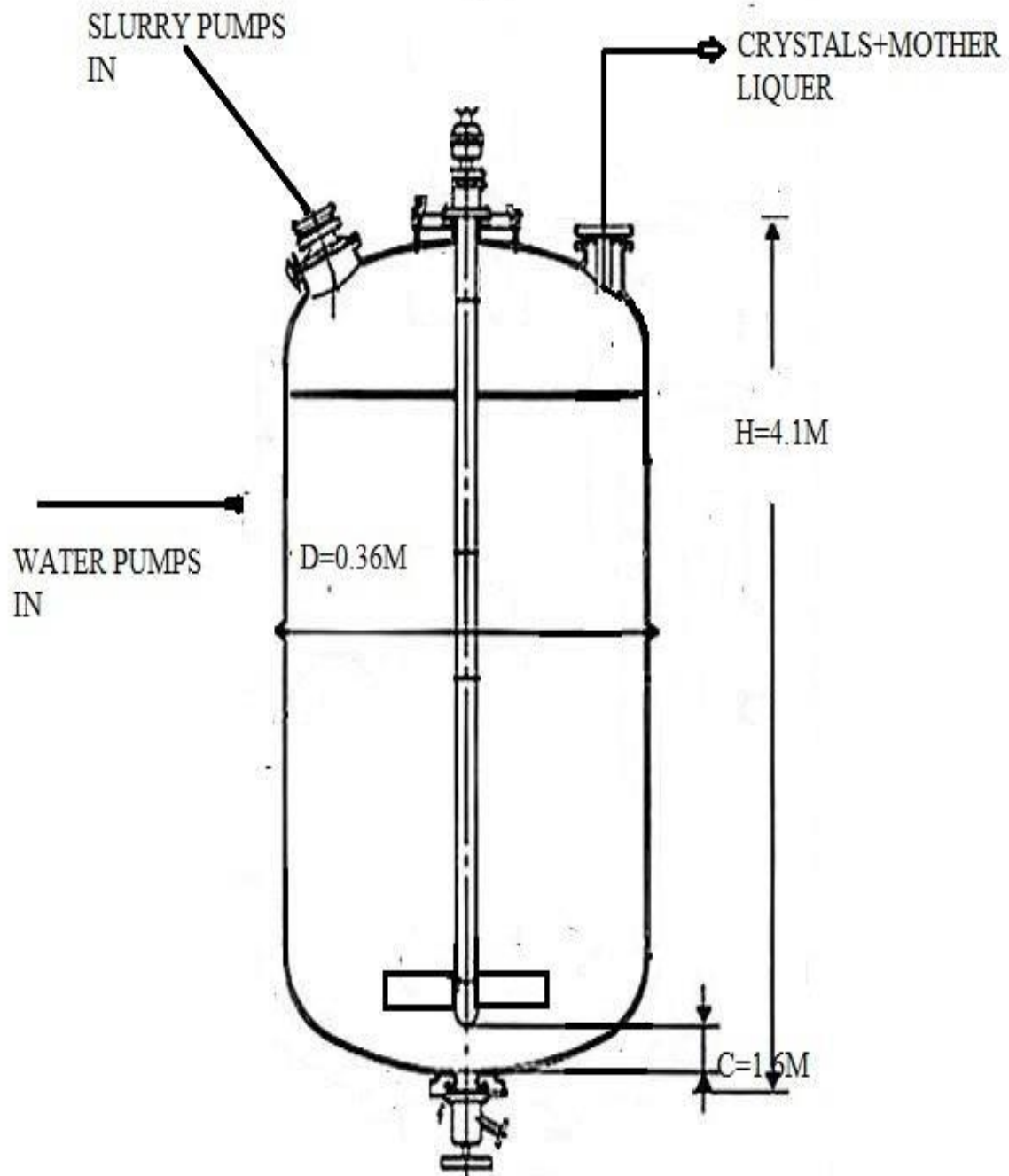


Fig6- design of crystallizer

CHAPTER 7

INTRUMENTATION & PROCESS CONTROL

7 INSTRUMATATION& PROCESS CONTROL

The most important application of instrumentation control is to achieve quality control of final product which is attained by close control over the basic parameters and process variable in plant .The instrumentation deals with the measuring& sensing the process variable designed such that it allows for maximum variation in the process conditions without affecting the control variables. Intimation& control system become essential whenever tolerance is low & deviations from specific condition seriously affect the final product. Thus savings in raw materials is assured by minimization of rejects, hence cost reduction is achieved.

7.1 INSTRUMATATION& PROCESS CONTROL OF THE CSTR

The mathematical model of this CSTR comes from balances inside the reactor. A jacket surrounding the reactor also has feed and exit streams. The jacket is assumed to be perfectly mixed and at lower temperature than the reactor. Energy passes through the reactor walls into jacket, removing the heat generated by reaction. The control objective is to keep the temperature of the reacting mixture T , constant at desired value .The only manipulated variables the coolant temperature.

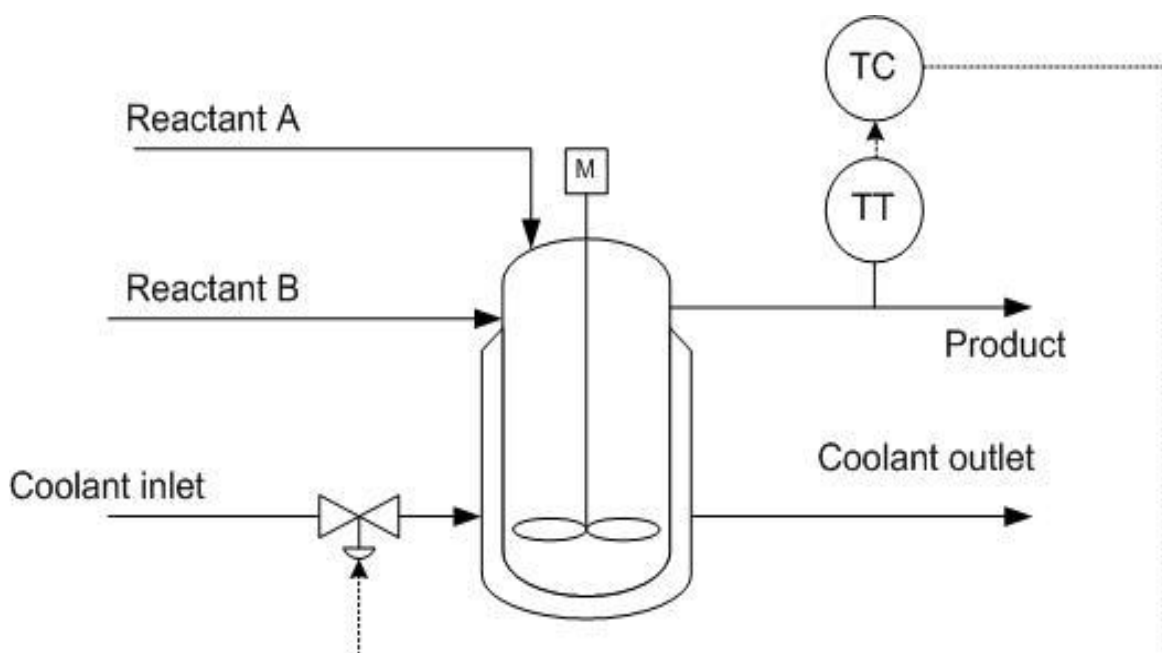


Fig 7- Instrumentation& process control of CSTR

7.2 INSTRUMATATION&PROCESS CONTROL OF CRYSTALLIZER

The crystallization apparatus employed in anti-solvent crystallization mode is composed of a subset of the entire apparatus built originally for Oxalic acid crystallization. The principal components include the crystallizer vessel with agitator, heat transfer coil , a conductivity meter ,an feed flask for the anti-solvent supply ; a variable speed peristaltic pump for anti-solvent addition , and a temperature-controlled circulating bath to heat or cool the contents of the crystallizer vessel through the heat transfer coil .

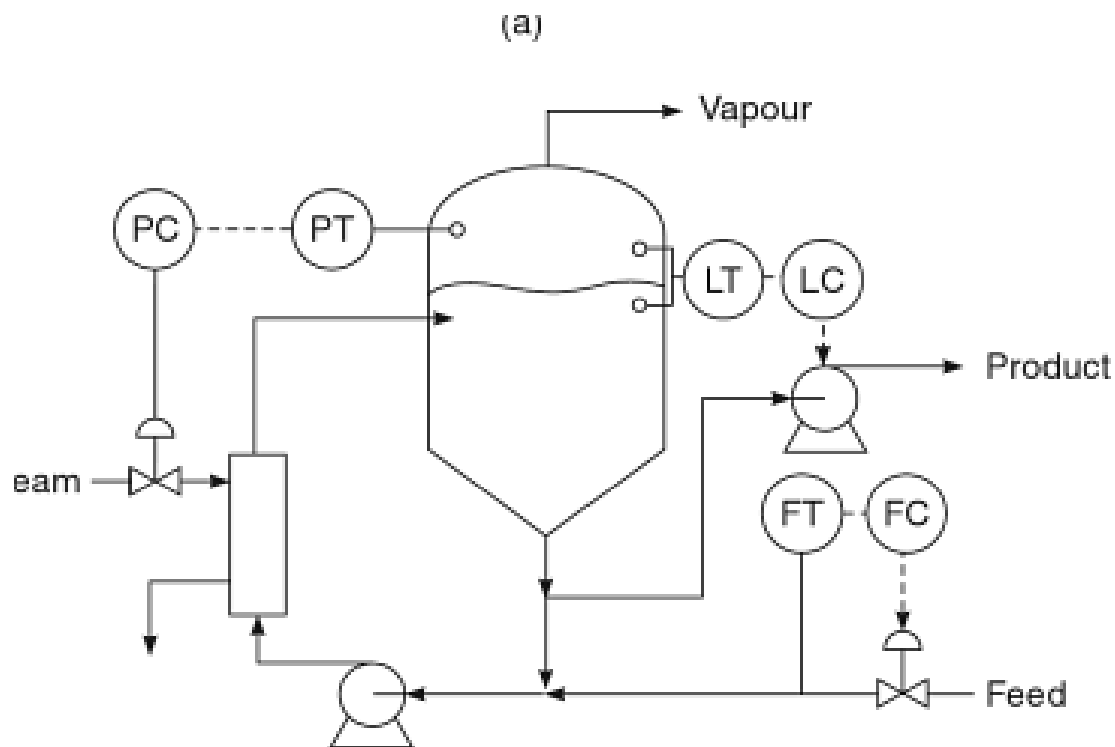


Fig 8- Instrumentation& process control of crystallizer

7.3 INSTRUMATATION & PROCESS CONTROL OF THE ABSOSBER

Reactants are taken from every section using custom-made storage tanks. These consist of special liquid collection rings which are machined into the section connectors. Pt100 temperature probes are inserted between every section. Additional temperature probes are placed at the inlets and outlets and in the recycled cleaned gas phase. Three pressure transmitters from Bugh&Bønsøe – the Tensto 6300 series – are placed in the bottom, middle, and top of the column, as indicated by PI1, PI6, and PI11. The gas phase composition is measured at the outlet of the column, Ci1. The Nitrogen dioxide transmitters used are Vaisala CARBOCAP® series GMT221. These cover a specified range, with a $\pm 2\%$ expected accuracy of the full scale reading. Flow is measured manually by four calibrated rotameters (F)

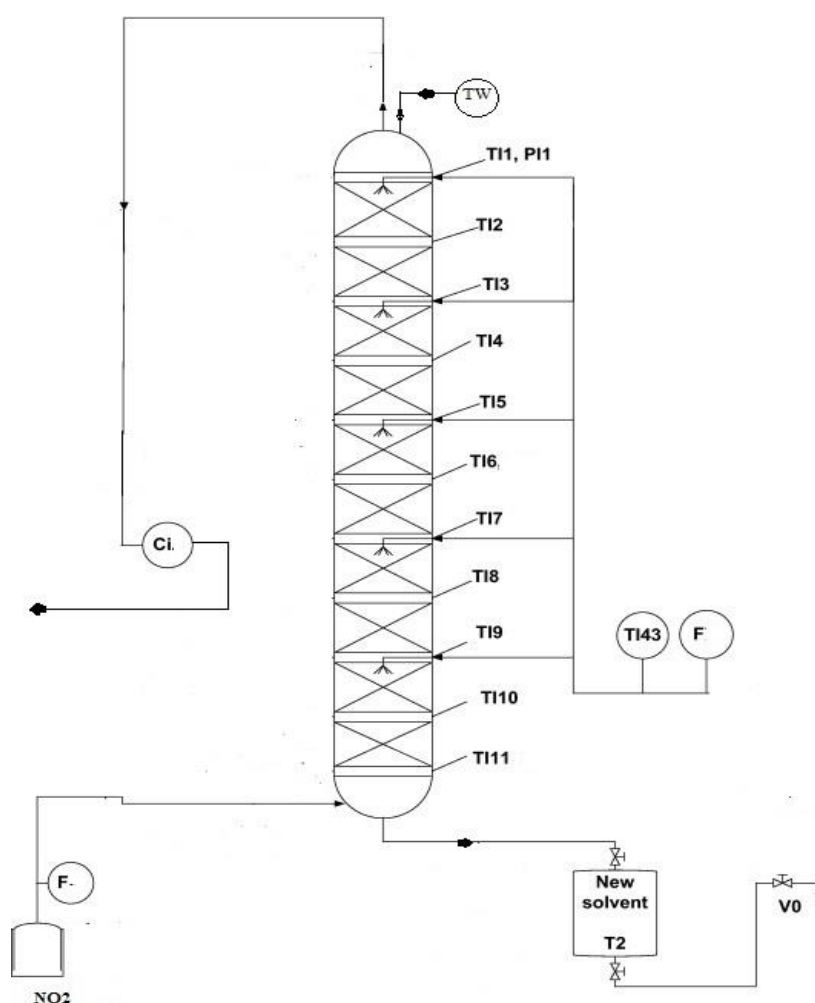


Fig 9- Instrumentation& process control of absorber

CHAPTER-8

PLANT ECONOMY

Cost estimation is an integral part for plant setup. Factors that should be consider for capital investment is given below. Fixed cost for plant (capital coat, building utility, process equipment, storage facilities etc.) Working capital like raw material process inventories, product, product, maintenance and repair works etc. The total production cost which includes manufacturing cost, raw material cost, operating cost, overhead cost, employee salary. Economic analysis which include selling price, income tax etc. According to report from combolchadedre in 2009

Average cost of oxalic acid plant set up was Rs. 7×10^8

Chemical Engineering Plant Cost Index:

Cost index in 1971 = 132

Cost index in 2002 = 402

Thus, Present cost of Plant = (original cost) * (present cost index)/(past cost index)

$$= (7 \times 10^8) * (402/132) = \text{Rs. } 21.3182 \times 10^8$$

i.e., Fixed Capital Cost (FCI) = Rs. 21.3182×10^8

Estimation of Capital Investment Cost:

I. Direct Costs: material and labour involved in actual installation of complete

Facility (70-85% of fixed-capital investment)

a) Equipment + installation + instrumentation + piping + electrical + insulation +

Painting (50-60% of Fixed-capital investment)

1. Purchased equipment cost (PEC): (15-40% of Fixed-capital investment)

Consider purchased equipment cost = 25% of Fixed-capital investment

$$\text{i.e., PEC} = 25\% \text{ of } 21.3182 \times 10^8 = 0.25 \times 21.3182 \times 10^8 = \text{Rs. } 5.32955 \times 10^8$$

2. Installation, including insulation and painting: (25-55% of Purchased equipment cost.)

The Installation cost = 40% of Purchased equipment cost

$$= 40\% \text{ of } 5.32955 \times 10^8 = 0.40 \times 5.32955 \times 10^8 = \text{Rs. } 2.13182 \times 10^8$$

3. Instrumentation and controls, installed: (6-30% of Purchased equipment cost.)

Consider the installation cost = 15% of Purchased equipment cost

$$= 15\% \text{ of } 5.32955 \times 10^8 = 0.15 \times 5.32955 \times 10^8 = \text{Rs. } 0.7994325 \times 10^8$$

4. Piping installed: (10-80% of Purchased equipment cost)

Consider the piping cost = 40% Purchased equipment cost

$$= 40\% \text{ of } 5.32955 \times 10^8 = 0.40 \times 5.32955 \times 10^8$$

$$= \text{Rs. } 2.13182 \times 10^8$$

5. Electrical, installed: (10-40% of Purchased equipment cost)

Consider Electrical cost = 25% of Purchased equipment cost

$$= 25\% \text{ of } 5.32955 \times 10^8 = 0.25 \times 5.32955 \times 10^8 = \text{Rs. } 1.3323875 \times 10^8$$

Hence total cost of (1+2+3+4+5) = 11.7250075×10^8 Rs. --- (54.99% of FCI)

B. Buildings, process and Auxiliary: (10-70% of Purchased equipment cost)

Consider Buildings, process and auxiliary cost = 40% of PEC

$$= 40\% \text{ of } 5.32955 \times 10^8 = 0.40 \times 5.32955 \times 10^8 = \text{Rs. } 2.13182 \times 10^8$$

C. Service facilities and yard improvement: (40-100% of Purchased equipment cost)

Consider the cost of service facilities and yard improvement = 62% of PEC

$$= 62\% \text{ of } 5.32955 \times 10^8 = 0.62 \times 5.32955 \times 10^8 = \text{Rs. } 3.304321 \times 10^8$$

D. Land: (1-2% of fixed capital investment or 4-8% of Purchased equipment cost)

Consider the cost of land = 5% of PEC = 5% of $5.32955 \times 10^8 = 0.05 \times 5.32955 \times 10^8$

$$= \text{Rs. } 0.2664775 \times 10^8$$

Thus, Direct cost = $\text{Rs. } 17.4276285 \times 10^8$ ----- (81.75% of FCI)

II. Indirect costs: expenses which are not directly involved with material and labour

Of actual installation of complete facility (15-30% of Fixed-capital investment)

A. Engineering and Supervision: (5-30% of direct costs)

Consider the cost of engineering and supervision = 15% of Direct costs

i.e., cost of engineering and supervision = 15% of 17.4276285×10^8

$$= 0.15 \times 17.4276285 \times 10^8 = \text{Rs. } 2.61414 \times 10^8$$

B. Construction Expense and Contractor's fee: (6-30% of direct costs)

Consider the construction expense and contractor's fee = 10% of Direct costs

i.e., construction expense and contractor's fee = 14% of 17.4276285×10^8

$$= 0.14 \times 17.4276285 \times 10^8 = \text{Rs. } 2.43986799 \times 10^8$$

C. Contingency: (5-15% of Fixed-capital investment)

Consider the contingency cost = 10% of Fixed-capital investment

i.e., Contingency cost = 10% of $17.4276285 \times 10^8 = 0.10 \times 17.4276285 \times 10^8$

$$= \text{Rs. } 1.74276 \times 10^8$$

Thus, Indirect Costs = $\text{Rs. } 6.796768 \times 10^8$ --- (29.88% of FCI)

III. Fixed Capital Investment:

Fixed capital investment = Direct costs + Indirect costs = $(17.4276285 \times 10^8) + (6.796768 \times 10^8)$

i.e., Fixed capital investment = $\text{Rs. } 24.2243965 \times 10^8$

IV. Working Capital: (10-20% of Fixed-capital investment)

Consider the Working Capital = 15% of Fixed-capital investment

i.e., Working capital = 15% of $24.2243965 \times 10^8 = 0.15 \times 24.2243965 \times 10^8$

$$= \text{Rs. } 3.19773 \times 10^8$$

V. Total Capital Investment (TCI):

Total capital investment = Fixed capital investment + Working capital

$$= (24.2243965 \times 10^8) + (3.19773 \times 10^8)$$

i.e., Total capital investment = $\text{Rs. } 27.42212 \times 10^8$

Estimation of Total Product cost:

I. Manufacturing Cost = Direct production cost + Fixed charges + Plant Overhead cost.

A. Fixed Charges: (10-20% total product cost)

i. Depreciation: (depends on life period, salvage value and method of

Calculation-about 10% of FCI for machinery and equipment, and 2-3%

For Building Value for Buildings)

Consider depreciation = 10% of FCI for machinery and equipment, and 3%

For Building Value for Buildings)

$$\begin{aligned}\text{i.e., Depreciation} &= (0.10 \times 21.3182 \times 10^8) + (0.03 \times 21.3182 \times 10^8) = \\ &= \text{Rs. } 2.771366 \times 10^8\end{aligned}$$

ii. Local Taxes: (1-4% of fixed capital investment)

Consider the local taxes = 4% of fixed capital investment

$$\text{i.e. Local Taxes} = 0.04 \times 21.3182 \times 10^8 = \text{Rs. } 0.852728 \times 10^8$$

iii. Insurances: (0.4-1% of fixed capital investment)

Consider the Insurance = 0.6% of fixed capital investment

$$\text{i.e. Insurance} = 0.006 \times 21.3182 \times 10^8 = \text{Rs. } 0.1279092 \times 10^8$$

iv. Rent: (8-12% fixed capital investment)

Consider rent = 10% of fixed capital investment

$$= 10\% \text{ of } 21.3182 \times 10^8$$

$$= 0.10 \times 21.3182 \times 10^8$$

$$\text{Rent} = \text{Rs. } 2.13182 \times 10^8$$

$$\text{Thus, Fixed Charges} = \text{Rs. } 5.8838232 \times 10^8$$

.

B. Direct Production Cost:

Now we have Fixed charges = 10-20% of total product charges – (given)

Consider the Fixed charges = 15% of total product cost

Total product cost = fixed charges/15%

Total product cost = $5.8838232 \times 10^8 / 15\%$

Total product cost = $5.8838232 \times 10^8 / 0.15$

Total product cost (TPC) = Rs. 39.225488×10^8

i. Raw Materials: (10-50% of total product cost)

Consider the cost of raw materials = 30% of total product cost

Raw material cost = 30% of $39.225488 \times 10^8 = 0.30 \times 39.225488 \times 10^8$

Raw material cost = Rs. 11.767646×10^8

ii. Operating Labour (OL): (10-20% of total product cost)

Consider the cost of operating labour = 15% of total product cost

Operating labour cost = 15% of $39.225488 \times 10^8 = 0.12 \times 39.225488 \times 10^8$

Operating labour cost = Rs. 5.883823×10^8

iii. Direct Supervisory and Clerical Labour (DS & CL): (10-25% of OL)

Consider the cost for Direct supervisory and clerical labour = 12% of OL

Direct supervisory and clerical labour cost = 12% of 5.883823×10^8

$= 0.12 \times 5.883823 \times 10^8$

Direct supervisory and clerical labour cost = Rs. 0.70606×10^8

iv. Utilities: (10-20% of total product cost)

Consider the cost of Utilities = 15% of total product cost

Utilities cost = 15% of $39.225488 \times 10^8 = 0.12 \times 39.225488 \times 10^8$

Utilities cost = Rs. 5.883823×10^8

v. Maintenance and repairs (M & R): (2-10% of fixed capital investment)

Consider the maintenance and repair cost = 5% of fixed capital investment

i.e. Maintenance and repair cost = $0.05 \times 21.3182 \times 10^8 = \text{Rs. } 1.06591 \times 10^8$

vi. Operating Supplies: (10-20% of M & R or 0.5-1% of FCI)

Consider the cost of Operating supplies = 15% of M & R

$$\text{Operating supplies cost} = 15\% \text{ of } 1.06591 \times 10^8 = 0.15 \times 1.06591 \times 10^8$$

$$\text{Operating supplies cost} = \text{Rs. } 0.1598865 \times 10^8$$

vii. Laboratory Charges: (10-20% of OL)

Consider the Laboratory charges = 15% of OL

$$\text{Laboratory charges} = 15\% \text{ of } 5.883823 \times 10^8 = 0.15 \times 5.883823 \times 10^8$$

$$\text{Laboratory charges} = \text{Rs. } 0.023982975 \times 10^8$$

viii. Patent and Royalties: (2-6% of total product cost)

Consider the cost of Patent and royalties = 5% of total product cost

$$\text{Patent and Royalties} = 5\% \text{ of } 39.225488 \times 10^8 = 0.05 \times 39.225488 \times 10^8$$

$$\text{Patent and Royalties cost} = \text{Rs. } 1.9612744 \times 10^8$$

$$\text{Thus, Direct Production Cost} = \text{Rs. } 35.10815288 \times 10^8$$

C. Plant overhead Costs (50-70% of Operating labour, supervision, and maintenance or 5-15% of total product cost); includes for the following: general plant upkeep and

overhead, payroll overhead, packaging, medical services, safety and protection, restaurants, recreation, salvage, laboratories, and storage facilities.

Consider the plant overhead cost = 60% of OL, DS & CL, and M & R

$$\text{Plant overhead cost} = 60\% \text{ of } ((5.883823 \times 10^8) + (0.70606 \times 10^8) + (1.60591 \times 10^8))$$

$$\text{Plant overhead cost} = 0.60 \times ((5.883823 \times 10^8) + (0.70606 \times 10^8) + (1.60591 \times 10^8))$$

$$\text{Plant overhead cost} = \text{Rs. } 7.655793 \times 10^8$$

Thus, Manufacture cost = Direct production cost + Fixed charges + Plant overhead costs.

$$\text{Manufacture cost} = 35.10815288 \times 10^8 + (5.883823 \times 10^8) + 7.655793 \times 10^8$$

$$\text{Manufacture cost} = \text{Rs. } 48.647769 \times 10^8$$

II. General Expenses = Administrative costs + distribution and selling costs + research and development costs

A. Administrative costs: (40-60% of operating labour)

Consider the Administrative costs = 50% of operating labour

$$\text{Administrative costs} = 0.5 * 5.883823 * 10^8$$

$$\text{Administrative costs} = \text{Rs. } 2.9419115 * 10^8$$

B. Distribution and Selling costs: (2-20% of total product cost); Includes costs for sales offices, salesmen, shipping, and advertising.

Consider the Distribution and selling costs = 10% of total product cost

$$\text{Distribution and selling costs} = 10\% \text{ of } 39.225488 * 10^8$$

$$\text{Distribution and selling costs} = 0.1 * 39.225488 * 10^8$$

$$\text{Distribution and Selling costs} = \text{Rs. } 3.9225488 * 10^8$$

C. Research and Development costs: (about 3% of total product cost)

Consider the Research and development costs = 3% of total product cost

$$\text{Research and Development costs} = 3\% \text{ of } 39.225488 * 10^8$$

$$\text{Research and development costs} = 0.03 * 39.225488 * 10^8$$

$$\text{Research and Development costs} = \text{Rs. } 1.17676 * 10^8$$

$$\text{Thus, General Expenses} = \text{Rs. } 8.04122703 * 10^8$$

III. Total Production cost = Manufacture cost + General Expenses

$$= (48.647769 * 10^8) + (8.04122703 * 10^8)$$

$$\text{Total production cost} = \text{Rs. } 56.6889893 * 10^8$$

IV. Gross Earnings/Income:

Wholesale Selling Price of oxalic acid per kg = £ 2.0

Let 1£ = Rs. 70.00

Hence Selling Price of acetaldehyde per kg = 2.0 * 70 = Rs. 140

Total Income = Selling price * Quantity of product manufactured

$$= 140 * (150 * 10^3 / \text{day}) * (330 \text{ days/year})$$

$$\text{Total Income} = \text{Rs. } 69.3 * 10^8$$

Gross income = Total Income – Total Production Cost

$$= (69.3 * 10^8) - (56.6889893 * 10^8)$$

$$\text{Gross Income} = \text{Rs. } 12.6110107 * 10^8$$

Let the Tax rate be 45% (common)

Taxes = 45% of Gross income

$$= 45\% \text{ of } 12.6110107 * 10^8 = 0.45 * 12.6110107 * 10^8$$

$$\text{Taxes} = \text{Rs. } 5.674954815 * 10^8$$

Net Profit = Gross income - Taxes = Gross income * (1 - Tax rate)

$$\text{Net profit} = 12.6110107 * 10^8 * (1 - 0.4) = \text{Rs. } 6.9360558 * 10^8$$

Rate of Return:

Rate of return = Net profit * 100 / Total Capital Investment

$$\text{Rate of Return} = 6.9360558 * 10^8 / (27.42212 * 10^8) = 0.2529 / 100 = 25.29\%$$

CHAPTER-9

PLANT LAYOUT

GENERAL GUIDELINES FOR PLANT LAYOUT-

This above process involves following steps

- (1) Analysis of entire plant process is done
- (2) Based upon that plant location is selected
- (3) When the plant location selection is done, the equipment associated for the plant is selected.

Note that all equipments have certain fixed guidelines for its setup (initial maintenance etc.)

Equipments for transport like conveyors and pipes are also selected based on the type of production. Distance between the equipments are maintained based upon its reactivity, emission etc. Ancillary buildings are setup based upon the employee and number of labours.

Specific ancillary building like workshop R& D lab which are associated with the plant are setup. Safety factor for each plant differs based on its production and process. So guidelines are installed for these. Crystallisers are kept in another sub section with boundaries. So that temperature generated by the reactors should not affect the cooling of the solution inside the crystallization. All the steam heaters should not face each other; it should be kept parallel to each other. Storage tank containing hazardous material should be at least 70 m distance from the side boundary. Now for the ancillary buildings they should be arranged so as to minimize the time spent by the personals from office building to plant. Near to gate first comes the pass division, then automobile stand. After that comes the administration building and purchase department. Workshop and fire and safety department are kept in other subsection and is near to the production plant. Hospital is located near to the plant in order to treat the cases of emergencies. Factors that must be considered when selecting a suitable site. are

- (1) Raw material supply

- (2)Transport facilities
- (3)Availabilty of water, fuel and power
- (4)Availability of cheap labour

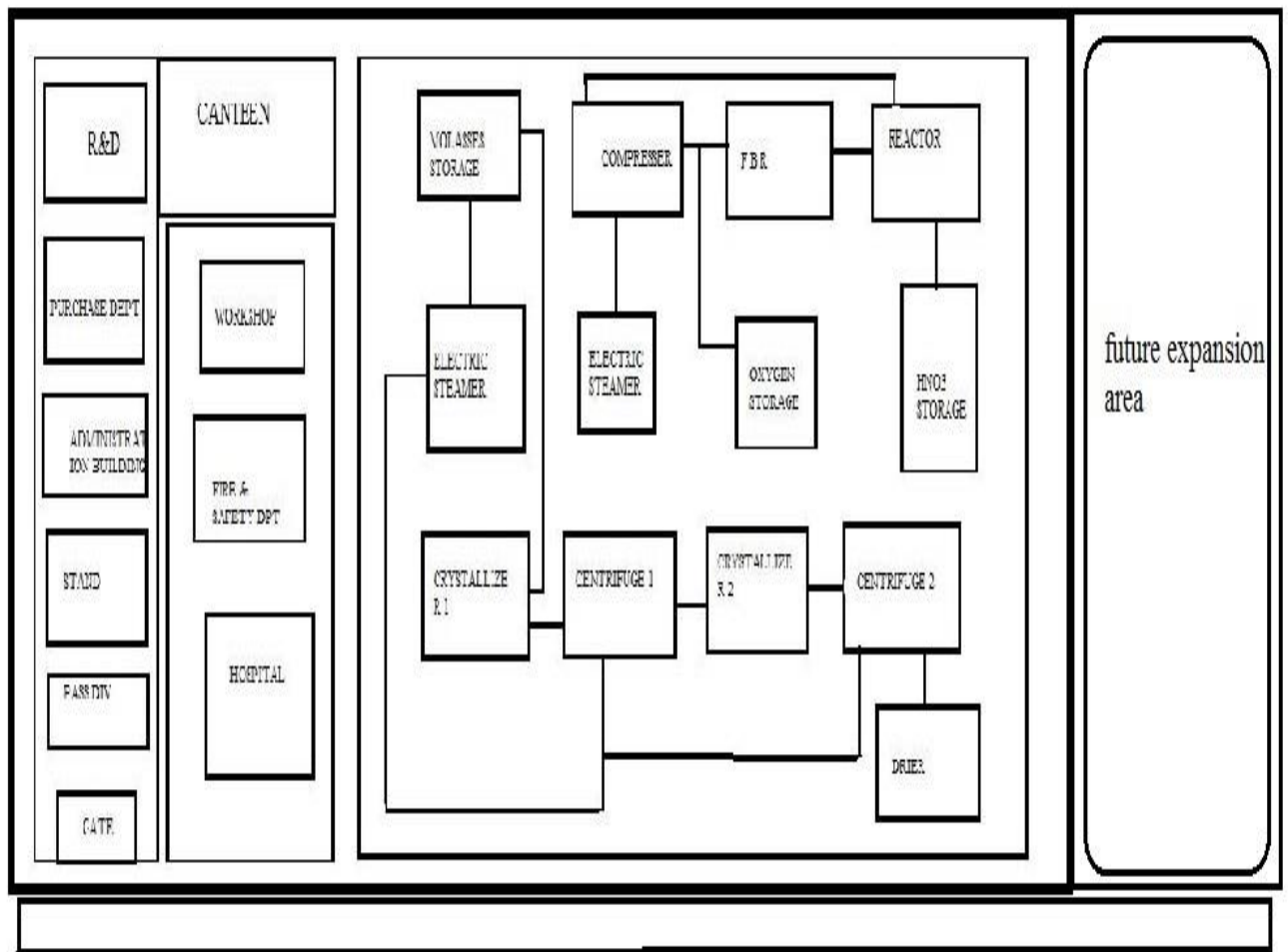


Fig 10- plant layout

CHAPTER 9

CONCLUSION

This above process lead to considerable improvement in the yield of oxalic acid in a single step .In this case, production was as high as 75% and also this process has an advantage of producing Nitric acid & Nitrogen- oxide gas which can be used in other industrial process or upon treatment of this by -products, it can also be used in the same plant. The purity of oxalic acid is 98%, which is best available in the market.

CHAPTER 10

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